ENVIRONMENTAL SCIENCE AND
TECHNOLOGY

OZONE AND THE DETERIORATION OF WORKS OF ART

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ABSTRACT

Seventeen artists' watercolor pigment samples and two Japanese woodblock prints were exposed to 0.40 ppm ozone in a controlled test chamber for three months. It was found that several artists' pigments when applied on paper will fade in the absence of light if exposed to an atmosphere containing ozone at the concentrations found in photochemical smog. Alizarin-based watercolors containing 1,2 dihydroxyanthraquinone lake pigments were shown to be particularly sensitive to ozone damage, as were the yellow pigments used in the Japanese woodblock prints tested. Indoor-outdoor ozone monitoring in a Pasadena, CA art gallery confirmed that ozone concentrations half as high as those outdoors can be found in art galleries that lack a chemically protected air conditioning system. Care should be taken to protect works of art from damage due to photochemical smog.

Introduction

The fading of pigments is a major hazard to works of art. Poor lightfastness usually is blamed, a process in which photochemical oxidation of the pigments occurs involving both light and oxygen (1-4). Short of encasing art objects in hermetically sealed inert atmospheres, the customary response has been to reduce lighting levels in museums and to restrict the display of light-sensitive artwork.

Ozone, an oxidant much stronger than oxygen, is formed by reactions between hydrocarbons and oxides of nitrogen in photochemical smog (5). In addition to its role as a respiratory irritant (6), ozone attacks organic materials, particularly at unsaturated double bonds. Recognized effects of ozone as an air pollutant include accelerated rubber cracking (7), fading of textile dyes (8-10), loss of tensile strength in fabrics (11), and erosion of exterior painted surfaces due to attack on the organic binder in the paint (12). The effect of ozone on the rate of deterioration of works of art is an open question.

In this study, the colorfastness of artists' pigments in the presence of ozone will be examined. It will be shown that several readily available artists' pigments when applied on paper are very sensitive to ozone damage, and will fade during a 95 day exposure to 0.40 ppm ozone in the absence of light.

Experimental Program

The fading of colorants depends on the entire system being studied: the pigments used, the substrate on which they are applied and the method of preparation (9). This experiment was designed specifically to examine the fading of pigments in preparations actually used by artists, but with little protection provided to the pigments by the presence of a heavy binder. For this purpose, pigments applied to paper with a very light binder were chosen for study: watercolors applied to watercolor paper, and Japanese woodblock prints.

Seventeen pigment samples were prepared by brushing dilute aqueous suspensions of Winsor and Newton artists' watercolors onto 5 cm by 10 cm strips of Bockingford watercolor paper (140 lb; pH 7). Pigments and their chemical composition are given in Table 1. The paper strips were cut in half to form two identical sets, one set for use in the ozone exposure experiment and the second set to be kept in a dark place as unexposed controls. Each batch of watercolor samples was accompanied by a section cut from a British blue scale. The British blue scale (British standard BS1006:1971) consists of eight pieces of wool dyed different shades of blue and rated in terms of permanence with respect to lightfastness. It is used by museums to detect conditions that pose a colorfastness hazard to artwork (13). The present experiment will determine whether the British blue scale also responds to fading due to ozone damage.

Two Japanese woodblock prints were acquired for inclusion in the ozone exposure experiment: a late edition struck from the blocks of a Hiroshige half plate Tokaido print of Minakuchi (print #1), and an early 20th century half plate reproduction of Minakuchi from the Hoeido Tokaido series by Hiroshige (print #2). Although neither print was an original, both were printed by traditional techniques, and microchemical tests (14-15) performed in our laboratories on print #2 showed that traditional pigments had been used (not necessarily the same pigments as in the originals, however).

The color of each sample was defined by hue, value and chroma using the Munsell System (16) of color notation. The hue describes a sample's relation to an equally spaced color wheel of ten major hues. The value is the lightness or darkness of a sample in relation to a neutral grey scale. Chroma indicates the departure of a given hue from a neutral grey of the same value. Munsell color matches were made under Sylvania Warmlight fluorescent lighting. Sample reflectance was determined in the red, green and blue using a Macbeth RD-517 reflectance microdensitometer. Reflectance relative to a barium sulfate white standard was measured continuously between 400 nm and 700 nm using a Varian Cary Model 219 reflectance spectrophotometer equipped with an integrating sphere. The sample spot viewed by the spectrophotometer does not coincide exactly with that seen by the microdensitometer.

High quality color photographic transparencies were taken of exposed and control samples before and after the experiment in order to

preserve a record of the condition of the samples in places where instrumental readings were not attempted. The photographic record was particularly important in the case of the woodblock prints. Those prints are unique, and it was not possible to maintain an unexposed control sample to document the condition of the prints before the experiment.

One set of samples was mounted onto backing sheets of watercolor paper using picture corners, then hung adjacent to the inside walls of a 55 liter light-tight stainless steel and glass ozone exposure chamber. Ambient air with temperature and humidity controlled by the laboratory air conditioning system was filtered to remove pre-existing pollutants. This air filtration system contained a packed bed of Purafil to remove SO_2 and NO_2, a packed bed of activated carbon to remove ozone and reactive hydrocarbons, and a particulate filter to eliminate any aerosol present. The filtered airstream was reozonated by an ultraviolet light source (Ultra-Violet Products Inc. Pen-ray lamp) and then pumped at a pressure slightly above 1 atm through the exposure chamber at a rate of approximately 2.2 l/min. A perforated tube was used to dispense the ozone upon introduction into the box, and the air volume within the chamber was stirred continuously. The ozone concentration within the box was measured continuously using a Dasibi model 1008-PC ozone monitor. The pigment samples and prints were exposed to 0.40 ± 0.08 ppm ozone at $23 \pm 2^{\circ}C$ and a relative humidity of 47 ± 8 % for 95 days. The error bounds stated represent one standard deviation of the ozone, temperature and relative humidity values

observed during the course of the experiment. Samples were withdrawn for observation at roughly monthly intervals. At the end of the experiment, color measurements on both the exposed samples and unexposed controls were repeated by the methods described previously.

As shown in Figures 1-4, several of the pigments tested faded dramatically during this experiment. Alizarin Crimson (sample 002) is a widely used red pigment prepared as a lake by precipitating 1,2 dihydroxyanthraquinone (Pigment Red 83, CI 58000) in an aluminum complex (17). The variety of metal complexes of alizarin that can be produced is discussed by Kiel and Heertjes (18-21), and the structure of the 1,2 dihydroxyanthraquinone molecule is compared to one proposed structure for an alizarin metal complex in Figure 5. Figure 1 shows that the ozone-exposed Alizarin Crimson sample reflects much more light in the blue and green than does the control, a result that the eye interprets as adding white light to the sample and giving a faded appearance. Crimson Lake (Figure 2) and Purple Lake (Figure 3) are close relatives to Alizarin Crimson, being lakes of 1,2 dihydroxyanthraquinone with the color modified by addition of other compounds (e.g. iron) during precipitation (23). The Crimson Lake faded considerably during the experiment, while changes in the Purple Lake were smaller.

An hypothesis can be advanced to explain the fading of the alizarin derivatives. Lebensaft and Salvin (24) have demonstrated that ozone attacks the anthraquinone textile dye Disperse Blue 3, breaking the anthraquinone ring system and forming products that include

phthalic acid. In this process, the chromophore is destroyed with a consequent loss of color. Although alizarin lake pigments are solids, their basic chemical structure is similar to Disperse Blue 3, suggesting that anthraquinone ring cleavage by ozone is the probable cause of rapid color loss in the alizarin-based artists' pigments in this experiment. Given the difference in the fading rate of Alizarin Crimson versus Purple Lake, it appears that details of the pigment formulations in addition to their 1,2 dihydroxyanthraquinone content affect the ozone-fastness of artists' pigments.

Ozone exposure may also produce a color shift (change in hue) if a coloring agent composed of a binary mixture of pigments of unequal colorfastness is used. The sample of Winsor and Newton's "Mauve" used in this experiment is a proprietary color blended from copper phthalocyanine and triphenylmethane lake (a lake of Basic Violet 14). As seen in Figure 4, the unexposed Mauve control sample shows reflectance peaks in both the red and blue yielding an initial shade that was purple. After exposure, the increase in reflectance is skewed toward the blue, and an observer would describe the exposed sample as a lighter blue shade. Apparently, the triphenylmethane lake component is fading, leaving the phthalocyanine blue behind. Triphenylmethane-based inks have been rapidly oxidized during lightfastness tests (1). It appears that ozone also will oxidize similar pigments in the dark.

Yellow pigments used in both of the Japanese woodblock prints faded noticeably. Although the colored areas in these prints are too small to be evaluated using the spectrophotometer, the

microdensitometer readings in Table 1 show a decline in color density, indicating that reflectance has increased, especially in the blue. Microchemical tests (14-15) on print 2 suggest that the yellow pigment used was either gamboge or turmeric. The chemical test employed makes use of the fact that gamboge and turmeric are both pH sensitive, and will change from yellow to orange when treated with a strong base (e.g. sodium hydroxide). Hence this test is not specific for either pigment. Both gambogic acid and curcumin (the major coloring agents in gamboge and turmeric, respectively) contain exposed carbon-carbon double bonds and substituted aromatic rings (22) that could be attacked by ozone (see Figure 5). It is interesting to note that the known gamboge watercolor pigment sample listed in the footnote to Table 1 did not change significantly during the course of this experiment. However, this does not eliminate the possibility that the yellow pigment used in the woodblock print was gamboge. Gamboge is a gum-resin mixture that can be prepared from the latex of any one of five species of the plant genus Garcinia (25). The powdered pigment produced in the orient was often adulterated with starch, flour or powdered resin (26). Thus the old pigments present in the woodblock prints could differ significantly from modern watercolor preparations, and hence may behave differently than modern pigment samples when exposed to ozone. In print 2, the foreground before ozone exposure was a dense blue green that subsequently faded (see Table 1). Microchemical tests (14-15) suggest that this pigment was a mixture of Prussian blue and either gamboge or turmeric.

The British blue scale exposed to ozone during this experiment did not change color or density in any of its eight dyed fabric sections. The British blue scale will not serve as a passive indicator of ozone-fastness problems in museums. The ozone exposure test ribbon designated for textile fading assessment by the American Association of Textile Chemists and Colorists (27) should be considered as a supplement to the British blue scale when monitoring for colorfastness problems in a museum environment.

Indoor Ozone Concentrations in Art Galleries

Previous comment on the subject of ozone exposure within museums is summarized by Thomson (28). He speculates that the organic material in antiquities might be damaged by ozone if ozone were present in museums, but then notes that the few indoor ozone concentration measurements made in London museums were "vanishingly low."

In order to determine whether or not high ozone concentrations can be observed in museums, a series of brief tests were conducted in Los Angeles area art galleries during the summer of 1982. The Baxter Art Gallery on the California Institute of Technology campus in Pasadena, California is a professionally managed gallery with shows often devoted to contemporary artists. Simultaneous indoor and outdoor ozone concentration measurements were made in Baxter Gallery on July 26, 1982 using a pair of Dasibi Model 1003 ozone monitors. Between 1320 hours and 1420 hours PDT an average concentration of 0.12 ppm O_3 was measured in the gallery at a time when the average outdoor O_3

concentration was at 0.20 ppm. This is consistent with the findings of others (29-31) that ozone concentrations inside institutional buildings and residences often are about half as high as those outdoors unless special precautions are taken.

Two larger art galleries in the Los Angeles area also were tested: the Huntington Gallery in San Marino, and the Los Angeles County Museum of Art in Los Angeles. Both of these museums were equipped with an activated carbon filter on the inlet to the building air conditioning system, a feature absent at Baxter Gallery. Ozone concentrations in these museums were very low, always below 0.01 ppm, confirming previous work (29) showing that activated carbon filters provide one means for preventing high indoor ozone concentrations.

Discussion

In this experiment, ozone exposure was found to affect a range of artists' pigments including organic yellows, a triphenylmethane/copper phthalocyanine-based mauve, and several anthraquinone lake reds and purples. While the natural organic yellows and triphenylmethane-based pigments have not previously been tested for ozone-fastness, the damage to the 1,2 dihydroxyanthraquinone lake pigments could have been expected based on previous work on related textile dyes (8,9,24,27). The results of the present experiment are not chemically surprising, but rather their importance lies in the context in which this ozone fastness problem is found. First, dispite the widespread reporting of the instability of textile dyes in the presence of ozone, the art

conservation community is generally unaware that a chemically similar problem occurs with certain artists' pigments. The change in medium from disperse dyes to pigment particles, and the use of trade names for pigments rather than their chemical structures may have helped to obscure this connection. Secondly, the potential for ozone damage to artwork presents a qualitatively different conservation problem than that encountered with commercial textiles. Consumer textiles usually are expected to perform well over a reasonably short service life (tens of years or less) while works of art in many cases must be preserved indefinitely. If the artists' pigments used in existing works of art or antiquities prove to be susceptible to ozone damage, it is too late to reformulate those pigments. Deliberate control of exposure to ozone would be the principal conservation procedure available for many existing works of art.

Previous experiments with the fading of textile dyes suggest that the rate of fading in the presence of ozone depends not only on the chemical nature of the dye, but also on (a) the fiber substrate into which the dye was absorbed, (b) the relative humidity during ozone exposure, and (c) the presence of oils or other materials mixed with the coloring agent that may absorb ozone hence increasing contact (9). The media used by artists vary significantly from practices in the textile industry, and the effect of these differences on the colorfastness of artwork should be investigated. In particular, watercolor pigments are composed of solid particles that rest on the surface of the paper used by the artist rather than being absorbed into

fabric fibers as is the case with textile dyes. Pigment size would be a major factor influencing the surface area of the coloring agent exposed to ozone in this case, and hence pigment size may influence the rate of reaction of watercolor pigments with ozone. Furthermore, artists' pigments are applied in the presence of various binders, ranging from a light gum base in the case of watercolors, to acrylic, alkyd or linseed oil bases in the case of thicker preparations. Whether these binders form a protective film over the pigment particles or alternatively absorb ozone thereby increasing contact should be investigated.

The atmospheric ozone concentration (0.40 ppm) used in the controlled chamber experiments in the present study is within the range of values observed in Los Angeles under heavy photochemical smog conditions. Annual mean ozone concentrations in Los Angeles are a factor of ten lower (32). As was demonstrated in Baxter Gallery and by other investigators (29-31) ozone concentrations inside buildings are often half as high as those outdoors unless precautions are taken, such as the use of activated carbon filters. Although it would take roughly three years (outdoors) to six years (in an unprotected building) to accumulate an ozone exposure (concentration times duration) from ambient air equivalent to the chamber experiments reported here, that duration is very short compared to the need to preserve works of art for hundreds of years. Ozone concentrations worthy of attention are not confined to Los Angeles; 538 counties in the United States are in violation of National Ambient Air Quality Standards for ozone (0.12 ppm

for one hour) (33). Given the existence of chemical processes leading to the destruction of artists' pigments by ozone and the possibility for transfer of significant outdoor ozone concentrations to some indoor environments, it is clear that atmospheric ozone could pose a threat to the preservation of works of art.

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Table 1. Chemical Composition of Pigments Tested, and Color Changes Noted after 95 Days of Exposure to 0.40 ppm Ozone. The criterion for a definite color change is an alteration of greater than ± 0.04 density units plus confirmation by a change in Munsell color match and spectrophotometer reading (if available). Possible change is indicated if that criterion is nearly met or if reason for suspecting a false positive test is known.

PIGMENT	CHEMICAL COMPOSITION	MUNSELL STANDARD		DENSITOMETER	
		Color before Exposure	Color after Exposure	Density before Exposure	Density after Exposure
		Hue Value/Chroma	Hue Value/Chroma	Red/Green/Blue	Red/Green/Blue
Watercolors(*)					
Definite change:					
002 Alizarin Crimson	1,2 Dihydroxyanthraquinone lake	10RP 5/14	10RP 6/12	0.28/1.00/0.91	0.21/0.70/0.65
017 Crimson Lake	1,2 Dihydroxyanthraquinone lake	8.75RP 6/12	5RP 8/6	0.22/0.74/0.61	0.13/0.38/0.31
030 Mauve	Triphenylmethane lake, plus copper phthalocyanine	2.5P 4/12	7.5B 6/8	0.79/0.97/0.58	0.69/0.67/0.39
Possible change:					
038 Purple Lake	1,2 Dihydroxyanthraquinone lake	10RP 6/8	10RP 6/8	0.35/0.75/0.71	0.32/0.69/0.66
Japanese Woodblock Print #1					
Definite change:					
Yellow (building panel)		2.5Y 8/6	7.5YR 7/4	0.23/0.38/0.58	0.22/0.35/0.45
Yellow/gray (building panel)		2.5Y 7/6	10YR 6/4	0.39/0.53/0.73	0.38/0.49/0.61
Japanese Woodblock Print #2(†)					
Definite change:					
Yellow (thatched roof)	Gamboge or turmeric (§)	10YR 8/6	10YR 8/4	0.21/0.34/0.53	0.21/0.31/0.45
Blue/green (foreground)	Prussian blue plus gamboge or turmeric (§)	7.5BG 4/2	2.5BG 5/2	1.02/0.92/0.89	0.89/0.77/0.79

(*) Watercolor pigments tested without showing definite change: Indian Red (natural and synthetic iron oxides); Rose Carthame (Bon arylamide and eosine lake); Carmine (cochineal); Lemon Yellow (barium chromate); Chrome Lemon (lead chromate and lead sulfate); Chrome Yellow (lead chromate); Gamboge (gum resin Natural Yellow 24); Vandyke Brown (humic earth with ferric oxide); Sap Green (chlorinated copper phthalocyanine; monoazo yellow lake plus a ferrous beta naphthol derivative); Hooker's Green Light (chlorinated copper phthalocyanine plus a ferrous beta naphthol derivative); Prussian Green (arylamide yellow and Prussian blue); Viridian (hydrated chromium sesquioxide); Cobalt Blue (cobalt aluminate or phosphate with alumina).

(†) Japanese woodblock print #2, possible color changes include: (1) Prussian Blue mountain area that is hard to measure because it is printed with a streaked wash; (2) an iron-containing red that may be overprinted onto yellow-containing foreground and thus is difficult to address separately; (3) a blue pigment used in people's clothing that has faded as shown by before and after photographs, but no densitometer data are available at that location in the print.

(§) Tentative identification.

LIST OF FIGURE CAPTIONS

Figure 1. Reflectance spectra of Alizarin Crimson watercolor pigment samples with (——) and without (—•—) exposure to 0.40 ppm O_3 for 95 days.

Figure 2. Reflectance spectra of Crimson Lake watercolor pigment samples with (——) and without (—•—) exposure to 0.40 ppm O_3 for 95 days.

Figure 3. Reflectance spectra of Purple Lake watercolor pigment samples with (——) and without (— —) exposure to 0.40 ppm O_3 for 95 days.

Figure 4. Reflectance spectra of Winsor and Newton's Mauve pigment blend samples with (——) and without (— —) exposure to 0.40 ppm O_3 for 95 days.

Figure 5. The chemical structure of several organic artists' pigments.

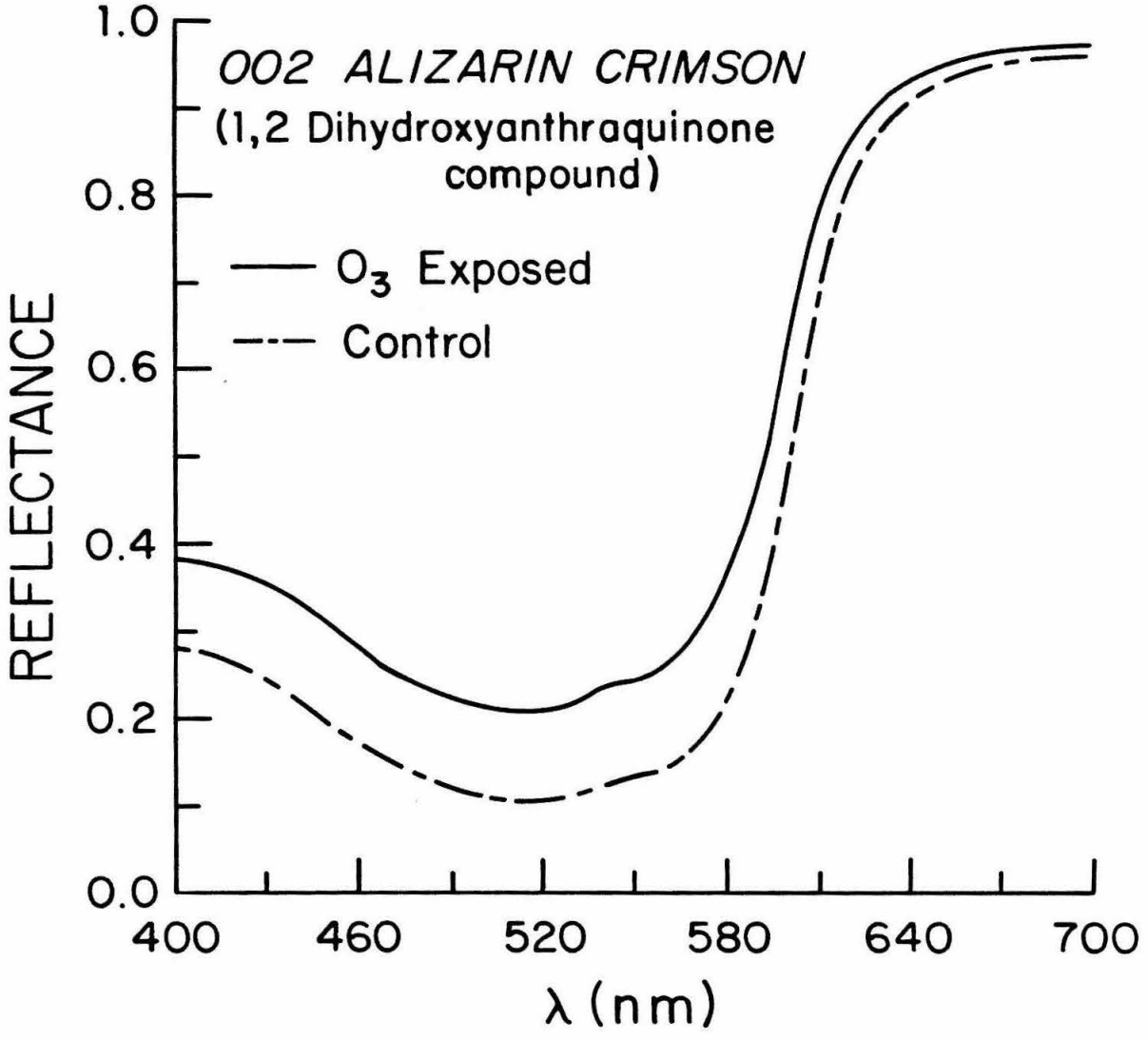

002 ALIZARIN CRIMSON
(1,2 Dihydroxyanthraquinone compound)
O_3 Exposed
Control
REFLECTANCE
1.0
0.8
0.6
0.4
0.2
0.0
400
460
520
580
640
700
λ (nm)

FIGURE 1

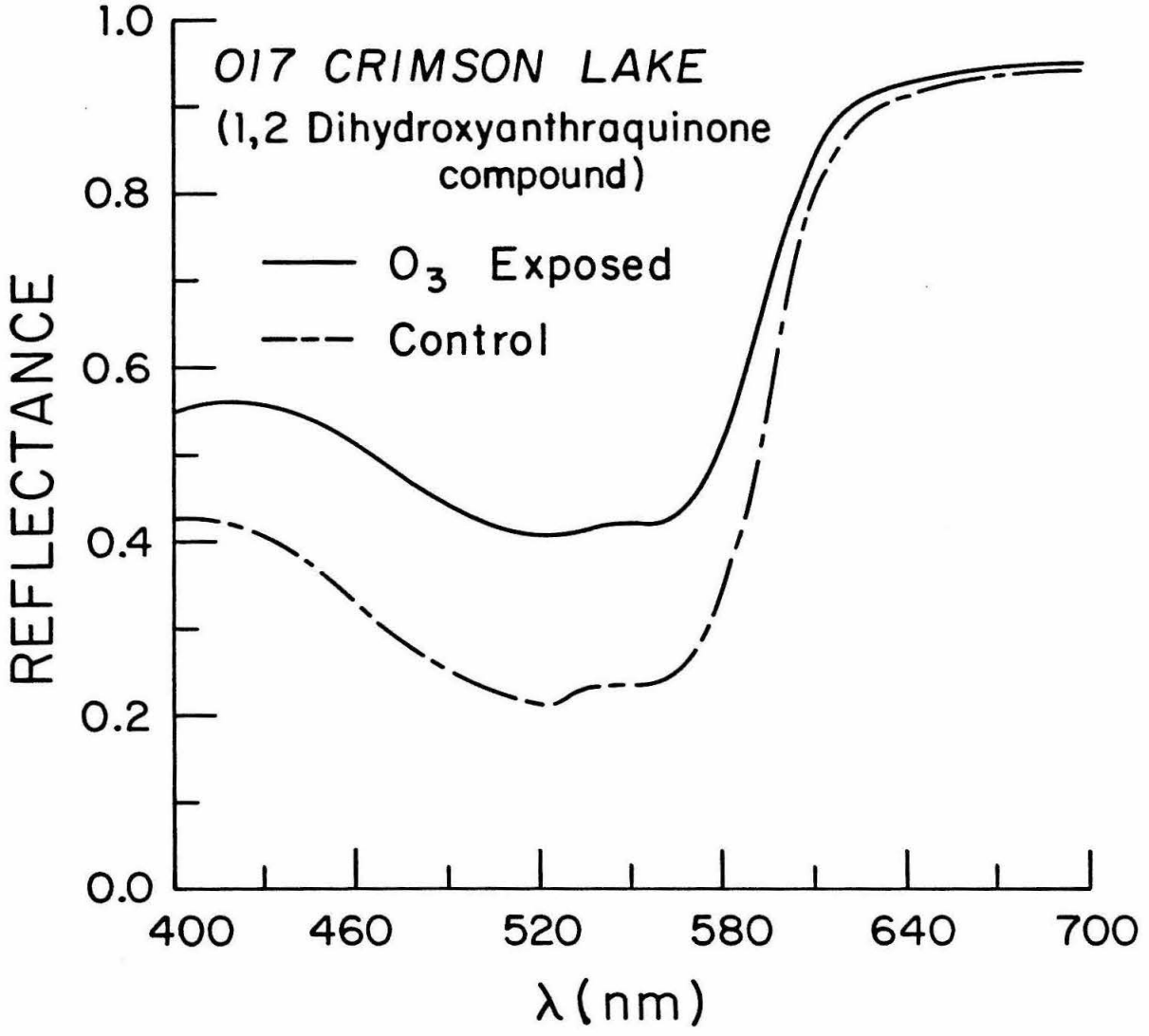

017 CRIMSON LAKE
(1,2 Dihydroxyanthraquinone compound)
O_3 Exposed
Control
REFLECTANCE
1.0
0.8
0.6
0.4
0.2
0.0
400
460
520
580
640
700
λ(nm)

FIGURE 2

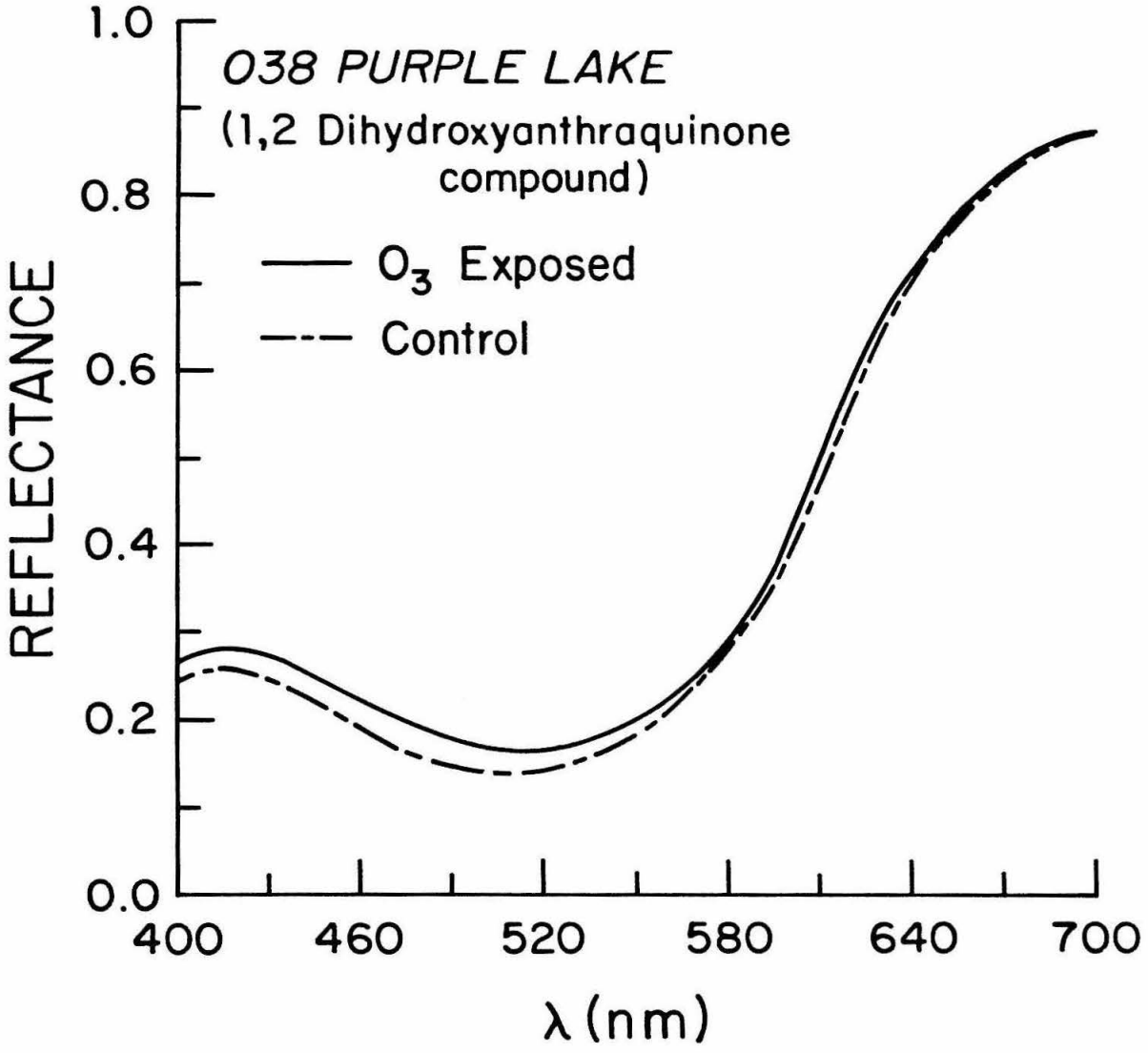

1.0
0.8
0.6
0.4
0.2
0.0
REFLECTANCE
038 PURPLE LAKE
(1,2 Dihydroxyanthraquinone compound)
O_3 Exposed
Control
400
460
520
580
640
700
λ (nm)

FIGURE 3

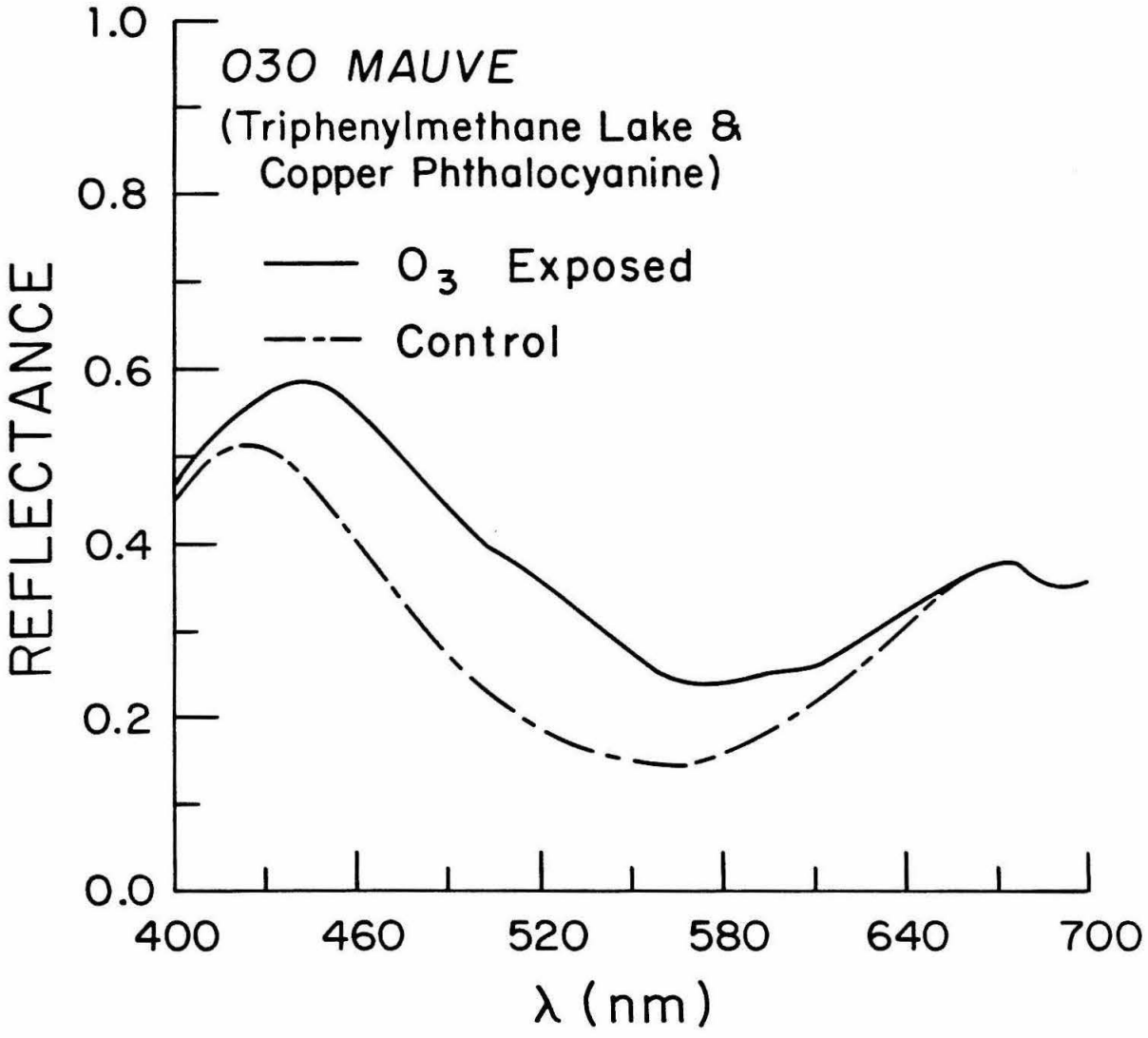
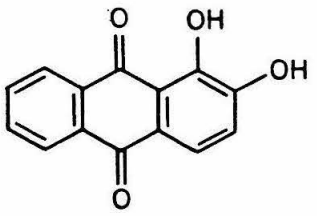
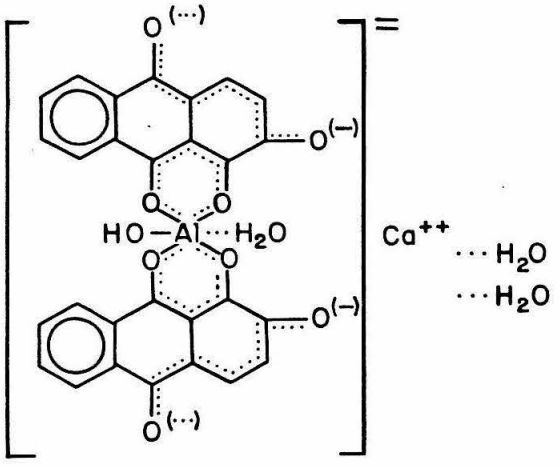

030 MAUVE
(Triphenylmethane Lake &
Copper Phthalocyanine)
O_3 Exposed
Control
REFLECTANCE
1.0
0.8
0.6
0.4
0.2
0.0
400
460
520
580
640
700
λ (nm)

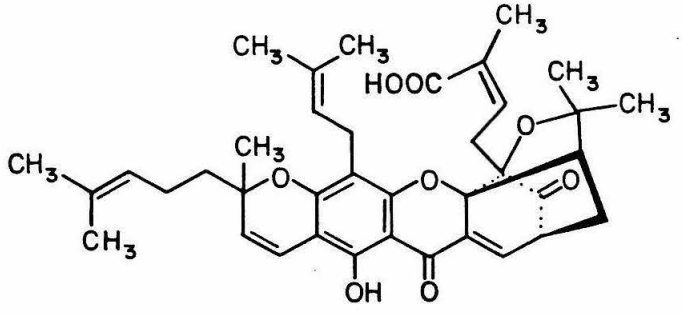
FIGURE 4



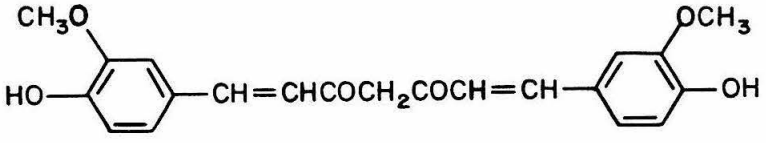
Alizarin
(1,2 dihydroxyanthraquinone)
Reference 22



An Alizarin Lake
(the calcium–aluminum lake shown as an example: $Ca \cdot Al(OH)(Az)_2 \cdot 3H_2O$)
References 18–21



Gambogic Acid
(the coloring agent in gamboge)
Reference 22



Curcumin
(the coloring agent in Turmeric)
Reference 22

FIGURE 5